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Analysis of Dry Sliding Wear Behaviour of Rice Husk Filled Epoxy Composites Using Design of Experiment and ANN

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Abstract

Artificial neural network (ANN) is a technique which can be used to simulate a wide variety of complex nonlinear engineering problems such as tribological performance of polymer composites. This article reports the implementation of ANN in analyzing the sliding wear performance of a new class of epoxy based composites filled with rice husk. Composites of four different compositions (5,10, 15 and 20 wt.% of rice husk reinforced in epoxy resin) are prepared in simple hand-lay-up technique. Physical, chemical and mechanical tests are conducted on these composites. Dry sliding wear experiments are conducted as per Taguchi's orthogonal array design. Significant control factors affecting specific wear rate are identified. Based on the data obtained from experiments, an ANN model is trained and tested to predict the effect of wear behaviour on various control factors. Factors like sliding velocity, filler content and normal load, in this sequence, are the significant factors affecting the specific wear rate. This work shows that rice husk possesses good filler characteristics, as it improves the sliding wear resistance of the polymer resin.

Key words: Dry sliding wear; Rice-husk; Epoxy; Taguchi design; ANN

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1. Introduction

Fiber reinforced polymer composites (FRPCs) have generated wide interest in various engineering fields including tribological applications such as cams, clutches, brakes, bearings, wheels, rollers, seals and gears due to their good combination of high specific strength, high modulus, low density and better wear resistance [1]. Being light weight they are the most suitable materials for weight sensitive uses, but their high cost sometimes becomes the limiting factor for commercial applications. Use of low cost, easily available fillers is therefore useful to bring down the cost of composites. Available literature suggests a large number of materials can be used as fillers in polymers [2]. The purpose of the use of filler can be divided into two basic categories; first to improve the mechanical, thermal and tribological properties and second, to reduce the cost of the component. Hard particulate fillers consisting of ceramic and metal particles and fiber fillers made of glass are being used these days to improve the tribological properties, even up to three orders of magnitude [3]. There have been various reports on the use of inorganic fillers like alumina and silica in polypropylene [4,5] and polyethylene [6,7]. But very few attempts have been made to utilize cheap material like rice husk, a bio waste, in preparing particle reinforced polymer composites.

A key feature of particle reinforced polymer composite that makes so promising as engineering materials is the opportunity to tailor the properties of material through the control of filler content and matrix combination. A judicious selection of matrix and the reinforcing solid particle phase can lead to a composite with a combination of strength and modulus comparable to or even better than those of conventional metallic materials [8]. Ceramic filled polymer composites have been the subject of extensive research in last two decades. The inclusion of ceramic fillers into polymers for commercial applications is primarily aimed at the cost reduction and stiffness improvement [9,10]. It has been reported that silica [11] plays an important role in improving electrical, mechanical and thermal properties of the composite. The shape, size, volume fraction and specific surface area of the added particles have been found to affect the mechanical properties of the composite greatly. Yamamoto et al.[12] have reported that the structure and shape of silica particle have significant effects on the tensile, fatigue and fracture properties of the composite. Few researchers like Satapathy and Patnaik [13] have used an industrial waste (red mud) as filler in analyzing the sliding wear performance of composites. Similarly, Kranthi and Satapathy [14] have used a bio waste (pine wood dust) as filler to study the wear performance of epoxy.

Against this background the present work is undertaken to investigate the influence of rice husk on wear resistance potential of epoxy resin. Rice husk is an inexpensive byproduct of human food processing and is considered as an agricultural waste, largely available from rice milling industries.

Because of large production of rice, approximately 600 million tons/year, there is a large amount of rice husk waste which is about 20 wt% of the total rice production [15]. The major components of rice husk are 32% cellulose, 21% hemicelluloses, 22% lignin and 15% mineral ash [16]. Rice husk has been used as a reinforcing material for recovery of used rubber tire in powder form by using sintering method [17]. Though many investigators have used rice husk as potential filler in various polymer matrices to study the mechanical, morphological and electrical properties [18-22] but there is no literature available on the study of wear performance of rice husk based polymer composites.

To study the correlation between the wear properties and the characteristic parameters is of prime importance for designing proper composites in order to satisfy functional requirements. In actual practice, the resultant wear rate is the combined effect of more than one interacting variable. In this regard, an expensive and easy to operate experimental strategy based on Taguchi's parameter design has been adopted to study the effect of various parameters and their interactions. This experimental procedure has been successfully applied for parametric appraisal in various wear process of a wide range of polymer matrix composites [23-27]. The present work also implements the artificial neural network technique to predict the wear rate of the epoxy based composites under different test conditions. A powerful ANN function is determined largely by the interconnections between the artificial neurons, similar to those occurring in their neural counterparts of biological systems. Prior to prediction of wear response in polymer composites, in this investigation, a certain amount of experimental results is required to train a well-designed neural network. After the network has learned to solve the material problems, new data from the similar domain can then be predicted without performing too many, long experiments.

Nomenclature

$\bar{\eta}_1$	Predicted average
$\bar{T}$	Overall experimental average
ρ_{ct}	Theoretical density of the composite in gm/cc
W_m	Weight fraction of the matrix in wt.%
W_p	Weight fraction of the particulate in wt.%
ρ_m	Density of the matrix in gm/cc
ρ_p	Density of the particulate in gm/cc
W_s	Specific wear rate in mm^3/N-m

Δ_m	Mass loss in gm
V_s	Sliding velocity in m/sec
F_n	Normal load in N

2. Experimental details

2.1 Matrix and filler materials

In the present work, epoxy resin (LY 556) is used as the matrix material and its common name is Bisphenol-A-Diglycidyl-ether and it chemically belongs to 'epoxide' family. The epoxy resin and the corresponding hardener (HY 951) are supplied by Ciba Geigy India Ltd. Epoxy is chosen primarily because its excellent dimensional, thermal stability and good corrosion resistance [28]. The filler, rice husk has low density of 0.12gm/cc, thermal conductivity of 0.095 W/m.K [29]. The husk is collected from Shiva Shakti Rice Mill, Dhenkanal, Odisha, India. The collected husk is dried in a woven with a temperature of 105^{0}C for removal of moisture and then sieved to an average particle size in the range of 90-100μm.

2.2 Composite fabrication

The low temperature curing epoxy resin and the corresponding hardener are mixed in a ratio of 10:1 by weight as recommended. Rice husk particles are reinforced in epoxy resin (density 1.1 gm/cc) to prepare the composites are listed in Table 1. The dough (epoxy filled with rice husk) is then slowly decanted into the glass tubes, coated before hand with wax and uniform thin film of silicone-releasing agent. The composites are cast by simple mechanical stirring and gradually poured in glass tubes (Borosil) so as to get cylindrical specimens of diameter 9mm and length 120mm.

2.3 Mechanical, Physical, chemical and micro-structural characterization

Micro-hardness measurement is done using a Leitz micro-hardness tester. A diamond shaped indentor, in the form of a right pyramid with a square base and an angle of 136^0 between opposite faces, is forced into the material under a load P. The two diagonals X and Y of the indentation left in the surface of the material after removal of the load are measured and their arithmetic mean d is calculated. In the present study, the load is equal to 2.5N and Vickers hardness number is calculated as:

$$H_V = 0.1889\frac{L}{d^2} \tag{1}$$

The tensile test is generally performed on flat specimens. The commonly used specimens are dog-bone type with end tabs. A uniaxial load is applied through both ends. The test is conducted as per

ASTM D628 M91 standard for particulate filled polymer composites. The length and width of the test specimen should be 180 and 20mm respectively. The tensile test is conducted on universal testing machine Instron 3369 and results are analyzed to calculate the tensile strength of the specimens.

Table 1. Designation and composition of composite specimens

Designation	Composition
C_1	95 wt.% epoxy + 5wt.% rice husk
C_2	90 wt.% epoxy + 10 wt.% rice husk
C_3	85 wt.% epoxy + 15 wt. % rice husk
C_4	80 wt.% epoxy + 20 wt.% rice husk

The theoretical density of composite materials in terms of weight fractions of different constituents can easily be obtained as per the following equations given by Agarwal and Broutman [30].

$$\rho_{ct} = \frac{1}{(W_p / \rho_p) + (W_m / \rho_m)} \tag{2}$$

Where, W and ρ represent the weight fraction and density respectively.

The suffix p, m and ct stand for the particulate, matrix and the composite sample respectively.

The raw rice husk is examined for the identification of the crystalline phases with a Philips X-Ray Diffractometer. The X-ray diffractogram is taken using Cu Kα radiation.

The surface morphology of the specimens are examined by the SEM JEOL JSM-6480LV. The worn out surfaces are mounted on stubs with silver paste. A thin film of platinum is vacuum evaporated onto the samples for better conductivity before the examination.

2.4 Dry sliding wear test

A pin-on-disc sliding wear test setup (supplied by DUCOM) is employed for evaluating the performance of the samples. The test is carried out as per the ASTM G99 standard [31] for dry sliding wear of polymer composites. The counter body is a disc made of hardened alloy steel with hardness value of 72 HRC and surface roughness (Ra) of 0.6 μm. The specimen is held stationary against the rotating steel disc and the normal force is applied through a lever mechanism as shown in Fig. 1. The sample is weighed in a precision electronic balance to an accuracy of ±0.1mg. The difference between the initial and final weights of the specimen is the mass loss under sliding. The specific wear rate can be defined as the volume of material removed per unit load per unit sliding distance and it can be expressed in terms of volume loss basis as:

$$W_s = \frac{\Delta m}{\rho t} V_s \cdot F_n \tag{3}$$

Where, Δm is the mass loss (gm), ρ is the density of the sample (gm/mm^3), t is the test duration (second), V_s is the sliding velocity (m/s) and F_n is the normal load (N).

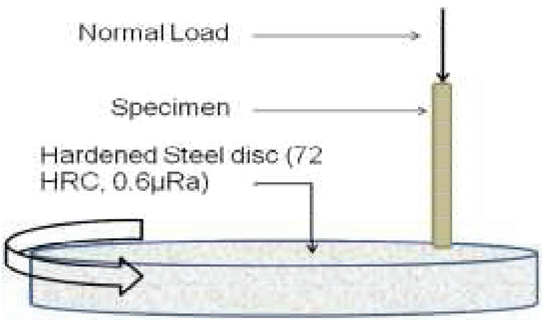


Fig. 1. Schematic diagram of pin-on-disc setup.

2.4.2 Experimental design

Design of experiment is a powerful analysis tool for modelling and analyzing the influence of control factors on performance output. The most important stage in the design of experiment lies in the selection of control factors. Therefore, a number of factors are included so that non-significant variables can be identified at the earliest opportunity. The wear test is carried out under operating conditions given in Table 2. Four control factors viz. sliding velocity, normal load, filler (rice-husk) content and sliding distance each at four levels are selected in accordance with L_{16} orthogonal array design. In the conventional full factorial design, 4^4=256 number of experiments are required to conduct for performance output where as in Taguchi design of experiment only 16 number of experiments are required, thus offering a greater advantage with respect to cost and time of experiment. The *S/N* ratio for minimum wear rate is coming under "Lower is better" (LB) characteristics and the logarithmic transformation of loss function is shown below as:

$$\frac{S}{N} = -10\log_{10}\left[\frac{1}{n}\left(\Sigma y^2\right)\right] \tag{4}$$

Where, n is the number of observations and y is the observed data.

The plan of experiment is: first column is assigned to sliding velocity (A), second column is assigned to normal load (N), third column is assigned to filler (rice-husk) content (wt.%) and fourth column is assigned to sliding distance (m).

Table 2. Levels of the variables used in the experiment

Control Factor	Level				
	I	II	III	IV	Units
A: Sliding velocity	100	200	300	400	cm/s
B: Normal load	5	10	15	20	N
C: Rice husk content	5	10	15	20	wt.%
D: Distance of sliding	1200	1600	2000	2400	m

3. Results and discussion

3.1 Physical, chemical and mechanical strength

To ascertain the various phases of the oxide ceramics present in the raw rice husk, the X-ray diffractogram is taken and is shown in Fig. 2. It exhibits distinct peaks which are assignable to various metal oxide phases such as quartz (SiO_2), anatase (TiO_2), corundum (Al_2O_3) and hematite (Fe_2O_3).

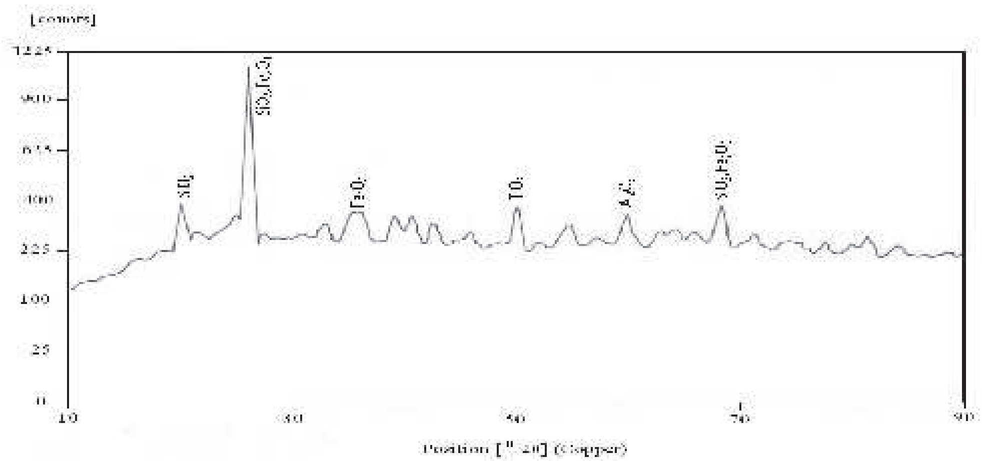


Fig. 2. X-ray diffraction of raw rice husk

Density is a material property which is of prime importance in several weight sensitive applications. The low densities of polymer composites are found to replace the conventional metals and materials in many engineering applications. Density of a composite depends on the relative weight proportion of matrix and the reinforcing components. A difference between the measured and the theoretical density values of a composite is observed due to the presence of voids and pores. These voids significantly affect the mechanical properties as well as the performance of composites. The theoretical and measured densities of the epoxy-rice husk composites are presented in Table 3. It has been observed that the density of the composite decreases with increase in filler content. The reduction of density is obvious as the true density of rice husk is about 0.1 times that of epoxy.

Table 3. Density values along with void fraction of the composites

Designation	Measured density (gm/cc)	Theoretical density (gm/cc)	Volume fraction of voids (%)
C_1	0.76	0.780	2.5
C_2	0.586	0.605	3.1
C_3	0.474	0.494	3.9
C_4	0.399	0.417	4.2

Hardness is considered as one of the most important factors that govern the wear resistance of any material. In the present work, micro-hardness values of the composites with rice husk fillers in different proportions have been obtained. It is observed that the hardness of the composites increases with increase in rice husk content. The micro-hardness values recorded in Vickers' scale for the composites are 28, 36, 39 and 41 Hv respectively.

The results for tensile strength are shown in Fig. 3. It is seen that in all the samples the tensile strength of the composites decreases with increase in filler content. The composite with 5wt.% of rice husk has strength of 174 MPa in tension and it is noticed that this value drops to 165.2 MPa with inclusion of 10wt.% of rice husk. The tensile strength of the sample is further drops to 154.3 MPa and 144.8 MPa in the case of other two composites with 15 and 20 wt.% of rice husk, respectively. Similar observations have been reported by Crespo et al. [18] for plasticized PVC composites and also by Yang et al. [20] for polypropylene matrix composites containing rice husk fillers. There may be two reasons for this reduction of tensile strength of the tested composite samples which can be explained as follows: (i) Due to poor chemical reaction between the rice husk particles and epoxy matrix, the interfacial bond strength becomes weak to carry the tensile load. (ii) Due to a rise of stress concentration at the sharp corners of irregular shaped rice husk particles embedded in the epoxy matrix.

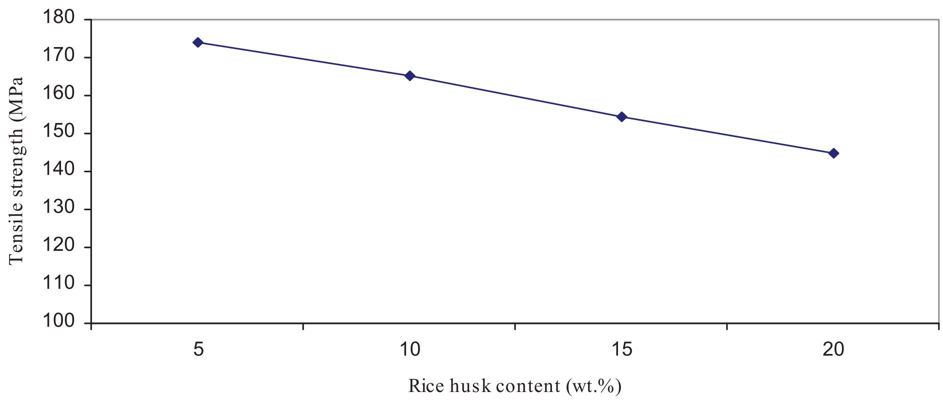
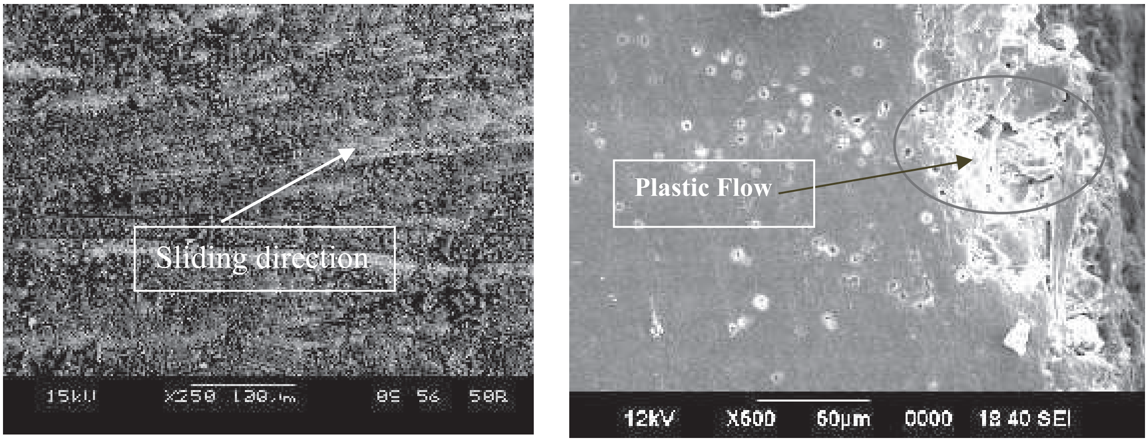


Fig. 3. Variation of tensile strength with rice husk content

3.2 Morphology of worn samples

The scanning electron micrographs of worn surfaces of rice husk filled epoxy composites with 15 wt.% are illustrated in Fig. 4(a) and(b). The micrographs have taken after 45 minute of the test duration with a sliding velocity of 300 cm/s under a normal load of 10N. The plastic flow of material in the sliding direction is clearly observed. It is due to the frictional heat generation between the composite sample and the hardened steel disc. The plastic flow of material increases with increase in sliding velocity and normal load. As a matter of fact, the rice husk particulates having sharp edge came out of the epoxy matrix and get aligned along the sliding direction and by virtue of their shape, size and hardness changes the wear resistance of the composite sample.



(a) (b)

Fig. 4. SEM micro-graphs of the worn composite surfaces

3.3 Wear analysis using experimental design

The specific wear rates obtained for all the sixteen test runs along with the corresponding S/N ratio are presented in Table 5. The overall mean S/N ratio is found to be -4.4168 dB. The values obtained by using MINITAB 14 software, specially used for design of experiment applications. The response analysis of all the factors for the S/N ratio is presented in Table 4. It is found that the sliding velocity is the most significant factor followed by filler content and normal load. Sliding distance is the least significant factor for specific wear rate of the composite samples under this study. The effects of individual factors are shown in Fig. 5. The analysis of the results leads to the conclusion that factor combination of A_1, B_3, C_4 and D_3 gives minimum wear rate in dry sliding situations.

3.3.1 Factor setting for minimum specific wear rate

For the present investigation, an attempt has been made to find out optimal setting of control factors for minimum specific wear rate. The single objective optimization requires quantitative determination of the relationship between specific wear rate and control factors. In order to derive the specific wear rate in terms of a mathematical model, the following equation is suggested:

$$W = K_0 + K_1 \times A + K_2 \times B + K_3 \times C \quad (5)$$

W is the specific wear rate in mm^3/N-m and K_i (i= 0,1,3) are the model constants. A is the sliding velocity (cm/s), B is the normal load (N) and C is the filler content (wt.%). The constants are calculated using non-linear regression analysis with the help of SYSTAT 7 software and the following relation is obtained:

$$W = 1.624 + 0.002 \times A - 0.014 \times B - 0.047 \times C \quad (6)$$

The correctness of the calculated constants is confirmed as high correlation coefficient (r^2) to the tune of 0.998 is obtained for specific wear rate and therefore the model is quite suitable to use for further analysis. A comparison of specific wear rate of the composites with the experimental values is presented in Table 6 and the percentage error varies between 0 to 13%, which lies within the acceptable limit.

Table 4. Signal to noise response table for specific wear rate

Level	A (cm/s)	B(N)	C(wt.%)	D(m)
1	-2.514	-5.270	-6.199	-4.532
2	-3.637	-4.651	-5.325	-4.368
3	-5.136	-3.862	-3.623	-4.171
4	-6.383	-3.888	-2.523	-4.599
Delta	3.869	1.408	3.676	0.429
Rank	1	3	2	4

Table 5. Experimental design using L_{16} orthogonal array

Test Runs (L_{16})	A (cm/s)	B (N)	C (wt%)	D (m)	Specific wear rate (mm^3/N-m)	S/N ratio
1	100	5	5	1200	1.801	-5.11027
2	100	10	10	1600	1.464	-3.31082
3	100	15	15	2000	1.127	-1.03848
4	100	20	20	2400	1.071	-0.59579
5	200	5	10	2000	1.878	-5.47391
6	200	10	5	2400	1.985	-5.95521
7	200	15	20	1200	1.123	-1.00760
8	200	20	15	1600	1.275	-2.11020
9	300	5	15	2400	1.795	-5.08129
10	300	10	20	2000	1.425	-3.07630
11	300	15	5	1600	2.147	-6.63664
12	300	20	10	1200	1.939	-5.75156
13	400	5	20	1600	1.865	-5.41358
14	400	10	15	1200	2.056	-6.26046
15	400	15	10	2400	2.179	-6.76514
16	400	20	5	2000	2.263	-7.09369

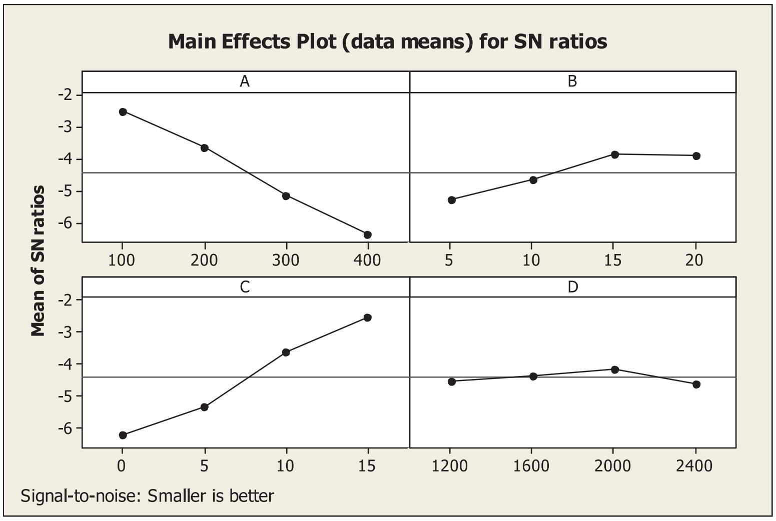


Fig. 5 Effect of control factors on sliding wear rate

3.3.2 Confirmation experiment

The confirmation experiment is the final test in the design of experiment process. The objective of the confirmation experiment is to validate the conclusions drawn during the analysis phase. The confirmation experiment is performed by conducting a new set of factor settings $A_3B_2C_2$ to predict the specific wear rate. A prediction equation can be formulated using Taguchi's approach to estimate S/N ratio for specific wear rate as [32]:

$$\bar{\eta}_1 = \bar{T} + (\bar{A}_3 - \bar{T}) + (\bar{B}_2 - \bar{T}) + (\bar{C}_2 - \bar{T}) \tag{7}$$

Where $\bar{\eta}_1$ is predicted average; $\bar{T}$ is overall experimental average; and $\bar{A}_3, \bar{B}_2$ *and* $\bar{C}_2$ are the mean responses for factors at designated levels. By combining like terms, the equation reduces to:

$$\bar{\eta}_1 = \bar{A}_3 + \bar{B}_2 + \bar{C}_2 - 2\bar{T} \tag{8}$$

A new arbitrary combination of factor levels A_3, B_2 and C_2 is used to predict the wear rate through prediction equation and the S/N ratio is found to be -6.2784 dB. An experiment was conducted under factor combination of A_3, B_2 and C_2 and the result was compared with the value obtained from predictive equation as shown in Table 7. The resulting model seems to be capable of predicting specific wear rate to a reasonable accuracy. An error of 4.85 % for S/N ratio of specific wear rate is observed. However, the error can further reduced if the number of measurement is increased. This validates the development of the mathematical model for predicting the measures of performance based on knowledge of the input parameters.

Table 6. Comparison of experimental and predictive equation results

Test run	Specific wear rate (mm^3/N-m)		Error (%)
	Result obtained from experiment	Result obtained from predictive equation	
1	1.801	1.754	2.6
2	1.464	1.449	1.02
3	1.127	1.144	1.50
4	1.071	0.939	12.32
5	1.878	1.720	8.41
6	1.985	1.884	5.08
7	1.123	1.109	1.24
8	1.275	1.274	0.07
9	1.795	1.684	6.18
10	1.425	1.379	3.22
11	2.147	2.014	6.19
12	1.939	1.709	11.86
13	1.865	1.649	11.58
14	2.056	1.814	11.77
15	2.179	1.979	9.17
16	2.263	2.144	5.25

Table 7. Results of the confirmation experiments for specific wear rate

	Optimal control parameters	
	Prediction	Experimental
Level	$A_3B_2C_2$	$A_3B_2C_2$
Specific wear rate(mm^3/N-m)	-6.2784	-5.9876

3.4 Wear Analysis and Prediction Using Neural Computation

The wear behaviour of composite materials is considered as a complex and non-linear problem with respect to its parameters and operating conditions [33]. In order to obtain minimum wear rate, appropriate combination of operating parameters have to be planned properly. The input variables are normalized so as to lie same range group of 0 to1. The output layer has one neuron to represent specific wear rate. About 75% of the experimental result data are used to train the network and different ANN structures with varying number of neurons in the hidden layer are tested at constant cycles, learning rate, error tolerance, momentum parameter, noise factor and slope parameter. Based on least error criterion, one structure is selected for training of input-output data (Table 8). The three layer neural network used in this work is shown in Fig. 6. The learning rate is varied in the range of 0.001-0.1 during training of input-output data. The number of neurons in the hidden layer is varied and optimized to 8. Software NEURALNET have been used for neural computation with back propagation algorithm and the specific wear rate is predicted within and beyond the experimental domain.

The ANN predictive results of specific wear rate for all the 16 test conditions are presented and compared with the experimental in Table 9. It is observed that the errors lie in the range of 0-10%, which establishes the validity of the neural computation. The errors, however, can still be reduced and the quality of predictions can be further improved by enlarging the datasets and by optimizing the construction of the neural network.

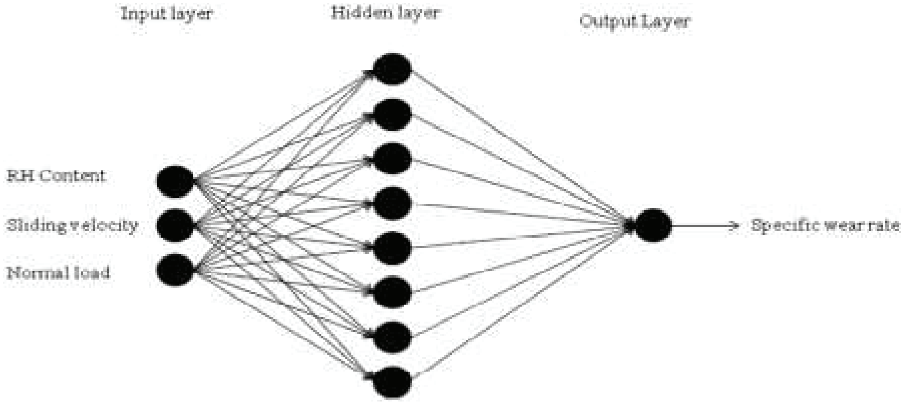


Fig. 6. The three layer neural network

Table 8. Selected input parameters for training of the neural network

Input parameters for Training	Values
Error tolerance	0.01
Learning rate (β)	0.002
Momentum parameter (α)	0.002
Noise factor (NF)	0.001
Number of epochs	10,00,000
Slope parameter (£)	0.6
Number of hidden layer neuron (H)	8
Number of input layer neuron (I)	3
Number of output layer neuron (O)	1

Table 9. Comparison of experimental and ANN results

Test run	Specific wear rate (mm^3/N-m)		Error (%)
	Result obtained from experiment	Result obtained from ANN prediction	
1	1.872	1.91	2.0
2	1.654	1.664	0.6
3	1.297	1.352	4.24
4	1.033	1.089	5.42
5	1.992	1.951	2.05
6	1.804	1.933	7.15
7	1.243	1.343	8.05
8	1.427	1.559	9.25
9	1.721	1.887	9.64
10	1.540	1.676	8.83
11	2.190	2.304	5.21
12	2.004	2.048	2.19
13	1.874	1.936	3.31
14	1.962	2.097	6.88
15	2.214	2.316	4.61
16	2.397	2.498	4.22

4. Conclusions

This experimental investigation into the sliding wear behaviour of rice husk filled epoxy matrix composites leads to the following conclusions.

1. Rice husk, an agricultural waste can be used as a potential filler material in epoxy matrix composites. A steady decline in the tensile strength is noticed in the filled composites whereas there is improvement in composite micro-hardness. The density of the composites is also greatly influenced by the filler content. Thus, while designing such composite systems for a specific requirement, there is a need for optimizing the rice husk content.
2. Dry sliding wear characteristics of these composites can be successfully analyzed using Taguchi experimental design scheme. This method provides a simple, systematic and efficient methodology for the optimization of the control factors.
3. Factors like sliding velocity, filler content and normal load in order of priority are the significant to minimize the specific wear rate.
4. Rice husk is found to possess good filler characteristics as it improves the wear resistance of the composite. SEM observations suggest that the presence of rice husk particles seems to have helped in restricting the mass loss from the composite surface due to sliding wear.
5. The use of neural network model to simulate experiments with parametric design is effective, efficient and helps to predict the sliding wear rate of rice husk-epoxy composites under different test conditions. The predicted and experimental values of wear rate shows good agreement and validates the remarkable capability of a well trained neural network.
6. In future, this study can be extended to polymer matrix composites using other filler materials.

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